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2-Nitrobenzyl methanesulfonate

Venkatramu Anuradha,^a S. Madan Kumar,^b
B. P. Siddaraju,^c N. K. Lokanath^b and P. Nagendra^{c*}^aDepartment of Physics, Dr M. G. R. Educational and Research Institute, Maduravoyal, Chennai, India, ^bDepartment of Studies in Physics, University of Mysore, Manasagangotri, Mysore 570 006, India, and ^cDepartment of Chemistry, BET Academy of Higher Education, Bharathi College, Bharathi Nagara, Mandya 571 422, India

Correspondence e-mail: nagendra088@yahoo.co.in

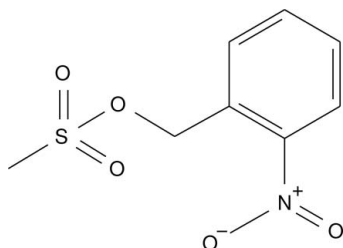
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.112; data-to-parameter ratio = 12.1.

In the title compound, $\text{C}_8\text{H}_9\text{NO}_5\text{S}$, the dihedral angle between the benzene ring and the nitro group is 5.86 (15°) and the $\text{C}-\text{O}-\text{S}$ group adopts an *anti* conformation [torsion angle = -168.44 (15°)]. In the crystal, molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, generating a three-dimensional network.

Related literature

For background to nitrobenzene derivatives, see: Ranu & Banerjee (2005); Ballini *et al.* (2005). For a related structure, see: Khan *et al.* (2008).



Experimental

Crystal data

 $\text{C}_8\text{H}_9\text{NO}_5\text{S}$
 $M_r = 231.23$ Monoclinic, $P2_1/c$
 $a = 12.414$ (3) Å $b = 7.967$ (2) Å
 $c = 10.994$ (3) Å
 $\beta = 112.235$ (11°)
 $V = 1006.5$ (5) Å³
 $Z = 4$ Cu $K\alpha$ radiation
 $\mu = 2.94$ mm⁻¹
 $T = 296$ K
 $0.23 \times 0.22 \times 0.21$ mm

Data collection

Bruker X8 Proteum CCD
diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2013)
 $T_{\min} = 0.552$, $T_{\max} = 0.578$ 6524 measured reflections
1663 independent reflections
1541 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.112$
 $S = 1.08$
1663 reflections138 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.41$ e Å⁻³
 $\Delta\rho_{\min} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C2}-\text{H2}\cdots\text{O1}^{\text{i}}$ | 0.93 | 2.54 | 3.266 (3) | 135 |
| $\text{C3}-\text{H3}\cdots\text{O2}^{\text{ii}}$ | 0.93 | 2.53 | 3.335 (3) | 145 |
| $\text{C7}-\text{H7B}\cdots\text{O4}^{\text{iii}}$ | 0.97 | 2.58 | 3.539 (3) | 169 |
| $\text{C8}-\text{H8A}\cdots\text{O4}^{\text{iii}}$ | 0.96 | 2.42 | 3.374 (4) | 172 |

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y + 1, z$; (iii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

We are grateful to the IOE, University of Mysore, for providing the single-crystal X-ray diffraction facility. PN thanks the BET Academy of Higher Education for research facilities.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7217).

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supporting information

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2-Nitrobenzyl methanesulfonate

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S1. Comment

Nitroalkenes have been used as substrates for Michael addition reactions (Ranu & Banerjee, 2005) and for the synthesis of many organic molecules (Ballini *et al.*, 2005).

The *ORTEP* of the title molecule is shown in figure 1. The molecules in the crystal structure are connected with C—H···O hydrogen bonds (Table 1). The C8—H8B···O4 hydrogen bond exhibits ring motifs of the type $R^2_2(8)$. The overall geometry of the title compound is similar to the 3,5-dinitrobenzyl methanesulfonate (Khan *et al.*, 2008). Overall packing of the molecule is shown in figure 2.

S2. Experimental

Red blocks were obtained from slow evaporation of a solution of ethanol.

S3. Refinement

The hydrogen atoms were fixed geometrically (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $= 1.2U_{\text{eq}}(\text{C})$.

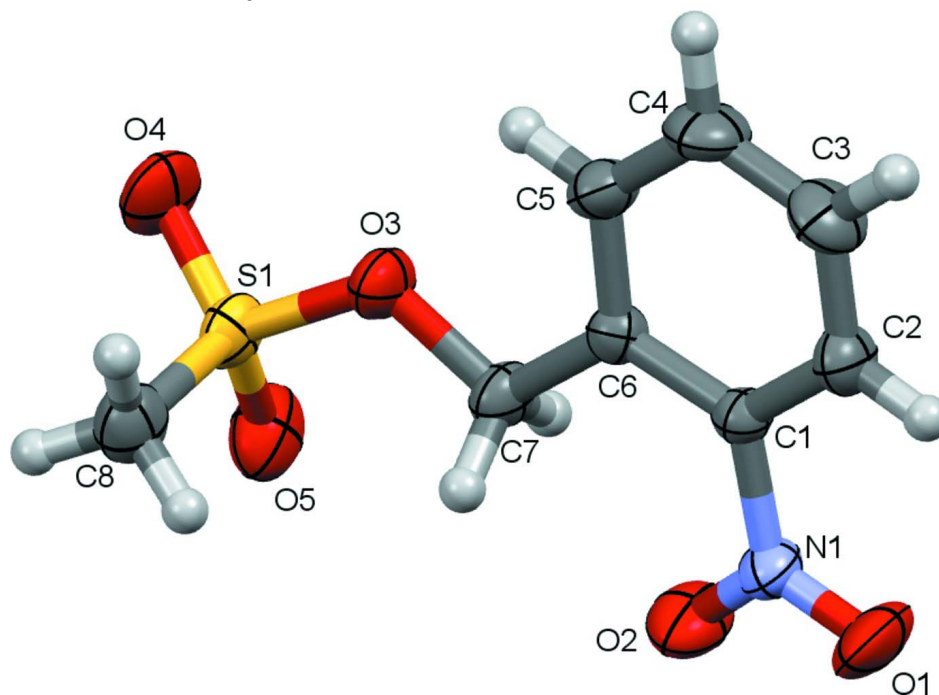
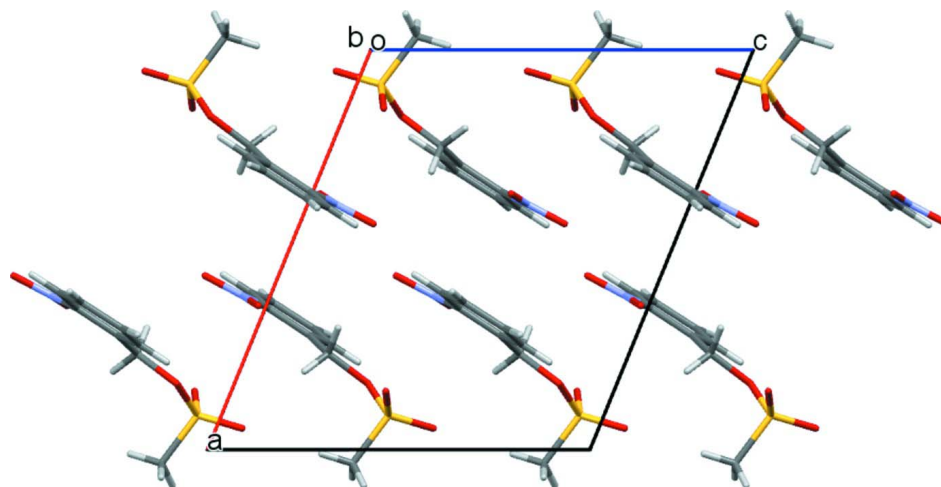


Figure 1

A view of the title molecule, with displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

A viewed along the *b* axis of the crystal packing of the title compound.

2-Nitrobenzyl methanesulfonate

Crystal data

$C_8H_9NO_5S$

$M_r = 231.23$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/bc$

$a = 12.414\ (3)\ \text{\AA}$

$b = 7.967\ (2)\ \text{\AA}$

$c = 10.994\ (3)\ \text{\AA}$

$\beta = 112.235\ (11)^\circ$

$V = 1006.5\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 480$

$D_x = 1.526\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 1663 reflections

$\theta = 3.8\text{--}64.5^\circ$

$\mu = 2.94\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, red

$0.23 \times 0.22 \times 0.21\ \text{mm}$

Data collection

Bruker X8 Proteum CCD

diffractometer

Radiation source: Bruker MicroStar microfocus

rotating anode

Helios multilayer optics monochromator

Detector resolution: $10.7\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2013)

$T_{\min} = 0.552$, $T_{\max} = 0.578$

6524 measured reflections

1663 independent reflections

1541 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 64.5^\circ$, $\theta_{\min} = 3.9^\circ$

$h = -14 \rightarrow 13$

$k = -8 \rightarrow 9$

$l = -12 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.112$

$S = 1.08$

1663 reflections

138 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from

neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.5912P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $FC^* = KFC[1 + 0.001XFC^2\Lambda^3/\text{SIN}(2\Theta)]^{-1/4}$
 Extinction coefficient: 0.0114 (9)

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|---------------|----------------------------------|
| S1 | 0.08637 (5) | 0.25179 (7) | 0.06271 (5) | 0.0405 (2) |
| O1 | 0.43500 (18) | 0.3906 (3) | 0.69206 (16) | 0.0672 (7) |
| O2 | 0.36003 (19) | 0.2381 (2) | 0.52088 (19) | 0.0601 (7) |
| O3 | 0.16548 (14) | 0.3941 (2) | 0.15092 (14) | 0.0458 (5) |
| O4 | 0.05351 (17) | 0.3133 (3) | −0.06740 (15) | 0.0592 (7) |
| O5 | 0.14617 (16) | 0.0958 (2) | 0.09607 (18) | 0.0584 (6) |
| N1 | 0.38559 (15) | 0.3744 (2) | 0.57357 (17) | 0.0376 (6) |
| C1 | 0.35397 (16) | 0.5255 (2) | 0.49184 (19) | 0.0295 (6) |
| C2 | 0.3940 (2) | 0.6755 (3) | 0.5560 (2) | 0.0411 (7) |
| C3 | 0.3686 (2) | 0.8231 (3) | 0.4862 (3) | 0.0523 (9) |
| C4 | 0.3040 (2) | 0.8185 (3) | 0.3534 (3) | 0.0533 (9) |
| C5 | 0.2634 (2) | 0.6678 (3) | 0.2901 (2) | 0.0424 (7) |
| C6 | 0.28708 (17) | 0.5157 (3) | 0.35717 (19) | 0.0310 (6) |
| C7 | 0.2405 (2) | 0.3532 (3) | 0.2870 (2) | 0.0387 (7) |
| C8 | −0.0341 (2) | 0.2504 (3) | 0.1059 (3) | 0.0526 (9) |
| H2 | 0.43780 | 0.67660 | 0.64580 | 0.0490* |
| H3 | 0.39490 | 0.92490 | 0.52830 | 0.0630* |
| H4 | 0.28730 | 0.91790 | 0.30560 | 0.0640* |
| H5 | 0.21920 | 0.66820 | 0.20030 | 0.0510* |
| H7A | 0.30420 | 0.28170 | 0.28830 | 0.0460* |
| H7B | 0.19630 | 0.29430 | 0.32990 | 0.0460* |
| H8A | −0.01050 | 0.22110 | 0.19700 | 0.0790* |
| H8B | −0.08950 | 0.16960 | 0.05320 | 0.0790* |
| H8C | −0.06920 | 0.35980 | 0.09150 | 0.0790* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|-------------|-------------|-------------|
| S1 | 0.0457 (4) | 0.0446 (4) | 0.0265 (3) | −0.0009 (2) | 0.0083 (3) | −0.0091 (2) |
| O1 | 0.0821 (13) | 0.0607 (13) | 0.0332 (10) | 0.0016 (10) | −0.0071 (9) | 0.0119 (8) |
| O2 | 0.0803 (13) | 0.0282 (9) | 0.0564 (12) | 0.0036 (8) | 0.0083 (10) | 0.0025 (7) |

| | | | | | | |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| O3 | 0.0555 (10) | 0.0432 (9) | 0.0289 (8) | −0.0069 (7) | 0.0050 (7) | −0.0037 (7) |
| O4 | 0.0670 (11) | 0.0806 (14) | 0.0262 (9) | −0.0037 (10) | 0.0134 (8) | −0.0061 (8) |
| O5 | 0.0595 (11) | 0.0463 (11) | 0.0559 (11) | 0.0051 (8) | 0.0065 (9) | −0.0161 (8) |
| N1 | 0.0376 (9) | 0.0337 (10) | 0.0345 (10) | 0.0023 (7) | 0.0056 (8) | 0.0053 (8) |
| C1 | 0.0322 (10) | 0.0256 (10) | 0.0298 (10) | 0.0008 (8) | 0.0106 (8) | 0.0013 (8) |
| C2 | 0.0463 (12) | 0.0365 (12) | 0.0349 (12) | −0.0059 (10) | 0.0090 (10) | −0.0070 (9) |
| C3 | 0.0668 (16) | 0.0272 (12) | 0.0591 (16) | −0.0085 (11) | 0.0197 (13) | −0.0070 (11) |
| C4 | 0.0712 (16) | 0.0295 (12) | 0.0571 (16) | −0.0019 (11) | 0.0220 (13) | 0.0121 (11) |
| C5 | 0.0531 (13) | 0.0382 (13) | 0.0325 (11) | −0.0006 (10) | 0.0123 (10) | 0.0058 (9) |
| C6 | 0.0339 (10) | 0.0299 (11) | 0.0303 (10) | −0.0002 (8) | 0.0135 (9) | −0.0014 (8) |
| C7 | 0.0465 (12) | 0.0361 (12) | 0.0279 (11) | −0.0004 (9) | 0.0079 (9) | −0.0034 (8) |
| C8 | 0.0529 (15) | 0.0635 (18) | 0.0402 (14) | −0.0060 (11) | 0.0163 (12) | −0.0060 (11) |

Geometric parameters (Å, °)

| | | | |
|----------|-------------|------------|-------------|
| S1—O3 | 1.5715 (17) | C4—C5 | 1.384 (3) |
| S1—O4 | 1.4181 (18) | C5—C6 | 1.391 (3) |
| S1—O5 | 1.4227 (18) | C6—C7 | 1.506 (3) |
| S1—C8 | 1.732 (3) | C2—H2 | 0.9300 |
| O1—N1 | 1.219 (2) | C3—H3 | 0.9300 |
| O2—N1 | 1.215 (2) | C4—H4 | 0.9300 |
| O3—C7 | 1.469 (3) | C5—H5 | 0.9300 |
| N1—C1 | 1.464 (2) | C7—H7A | 0.9700 |
| C1—C2 | 1.381 (3) | C7—H7B | 0.9700 |
| C1—C6 | 1.399 (3) | C8—H8A | 0.9600 |
| C2—C3 | 1.374 (3) | C8—H8B | 0.9600 |
| C3—C4 | 1.375 (4) | C8—H8C | 0.9600 |
| O3—S1—O4 | 104.31 (11) | O3—C7—C6 | 107.68 (18) |
| O3—S1—O5 | 109.14 (10) | C1—C2—H2 | 120.00 |
| O3—S1—C8 | 103.78 (11) | C3—C2—H2 | 120.00 |
| O4—S1—O5 | 119.00 (13) | C2—C3—H3 | 120.00 |
| O4—S1—C8 | 109.28 (14) | C4—C3—H3 | 120.00 |
| O5—S1—C8 | 110.13 (12) | C3—C4—H4 | 120.00 |
| S1—O3—C7 | 118.41 (14) | C5—C4—H4 | 120.00 |
| O1—N1—O2 | 122.6 (2) | C4—C5—H5 | 119.00 |
| O1—N1—C1 | 118.59 (18) | C6—C5—H5 | 119.00 |
| O2—N1—C1 | 118.77 (17) | O3—C7—H7A | 110.00 |
| N1—C1—C2 | 115.91 (17) | O3—C7—H7B | 110.00 |
| N1—C1—C6 | 121.17 (17) | C6—C7—H7A | 110.00 |
| C2—C1—C6 | 122.93 (18) | C6—C7—H7B | 110.00 |
| C1—C2—C3 | 119.6 (2) | H7A—C7—H7B | 108.00 |
| C2—C3—C4 | 119.3 (2) | S1—C8—H8A | 109.00 |
| C3—C4—C5 | 120.8 (2) | S1—C8—H8B | 110.00 |
| C4—C5—C6 | 121.7 (2) | S1—C8—H8C | 110.00 |
| C1—C6—C5 | 115.73 (19) | H8A—C8—H8B | 109.00 |
| C1—C6—C7 | 123.31 (19) | H8A—C8—H8C | 109.00 |
| C5—C6—C7 | 120.95 (18) | H8B—C8—H8C | 109.00 |

| | | | |
|-------------|--------------|-------------|------------|
| O4—S1—O3—C7 | −163.22 (18) | C2—C1—C6—C5 | 0.5 (3) |
| O5—S1—O3—C7 | −35.1 (2) | C2—C1—C6—C7 | −178.4 (2) |
| C8—S1—O3—C7 | 82.36 (19) | N1—C1—C6—C7 | 1.5 (3) |
| S1—O3—C7—C6 | −168.44 (15) | C1—C2—C3—C4 | −0.2 (4) |
| O1—N1—C1—C2 | 6.4 (3) | C2—C3—C4—C5 | 0.8 (4) |
| O2—N1—C1—C2 | −174.7 (2) | C3—C4—C5—C6 | −0.7 (4) |
| O2—N1—C1—C6 | 5.4 (3) | C4—C5—C6—C1 | 0.1 (4) |
| O1—N1—C1—C6 | −173.6 (2) | C4—C5—C6—C7 | 179.0 (2) |
| C6—C1—C2—C3 | −0.4 (4) | C1—C6—C7—O3 | 174.4 (2) |
| N1—C1—C6—C5 | −179.6 (2) | C5—C6—C7—O3 | −4.4 (3) |
| N1—C1—C2—C3 | 179.7 (2) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C2—H2 \cdots O1 ⁱ | 0.93 | 2.54 | 3.266 (3) | 135 |
| C3—H3 \cdots O2 ⁱⁱ | 0.93 | 2.53 | 3.335 (3) | 145 |
| C7—H7B \cdots O4 ⁱⁱⁱ | 0.97 | 2.58 | 3.539 (3) | 169 |
| C8—H8A \cdots O4 ⁱⁱⁱ | 0.96 | 2.42 | 3.374 (4) | 172 |

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $x, y+1, z$; (iii) $x, -y+1/2, z+1/2$.